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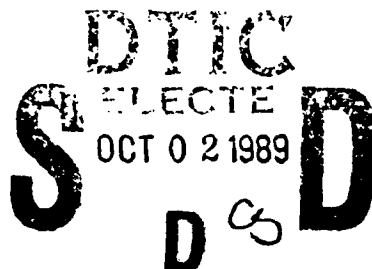
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STUDIES OF AFLAS FLUOROELASTOMERS

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July 1989



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INTRODUCTION

Aflas is the registered trademark for a group of fluoroelastomers manufactured in Japan* and marketed in the United States.† The chemical structure of Aflas is shown in Table 1. Only Aflas, Fluorel, and Viton, which are fluorocarbon elastomers, will be considered in this report. This table includes elastomers which are sold in bulk and does not include Kalrez elastomer parts, which are marketed by Du Pont. While Viton and Fluorel specialty elastomers are prepared from only fluorinated monomers, Aflas incorporates propylene to the extent of nearly 50% of the monomers polymerized, the balance being tetrafluoroethylene. Nuclear magnetic resonance spectroscopy (^{19}F) indicates that the polymer is at least 70 percent alternating, and that the methyl side groups are randomly arranged, resulting in an amorphous material.¹ Polymerization is carried out by emulsion polymerization using an ammonium persulfate-sodium bisulfite redox catalyst or another patented catalyst of the same type. The reactivity ratios for the monomers, $r_1(\text{C}_2\text{F}_4)$ and $r_2(\text{C}_3\text{H}_6)$ were calculated by the Fineman-Ross method² to be 0.05 and 0.10, respectively. If both r_1 and r_2 are much smaller than 1, this indicates low rate constants for the reactions of each monomer with itself at the polymer chain end. A high degree of alternation in the polymer results. The alternating character is undoubtedly responsible for the properties of the material, comparable in many respects to those of fluoroelastomers containing only fluorinated monomers. It is interesting to note that a copolymer of tetrafluoroethylene and ethylene is also commercially available under the trade name Tefzel, marketed by Du Pont. It is a thermoplastic and not an elastomer, which means that it is semicrystalline. The different types of Aflas and related polymers are shown in Table 2. In this report, a comparison of the properties of Aflas, Viton, and Fluorel will be made using available product literature. Then, characterization of the Aflas gumstock will be presented and, finally, comparison of the properties of an Aflas compound with a Fluorel compounded elastomer, including resistance to chemical agent simulants.

COMPARISON OF PROPERTIES

Fluorocarbon elastomers have excellent properties compared to other elastomers, especially in the areas of chemical resistance and high temperature use. Improvements in properties of the commercially important fluoropolymers have occurred by incremental improvements in polymerization, work up, and cure systems are not by development of new polymers.³ Since they might be compared for the same application, the properties of Aflas, Viton, and Fluorel, as they are given by the manufacturer, are listed in Tables 3, 4, and 5.

*Asahi Glass Co., Ltd., R & D Division, 1150 Hazawa, Kanagawa-Ku, Yokohama, Japan.

†Xenox, Inc., P.O. Box 79773, Houston, TX 77279.

1. KOJIMA, G., and KOJIMA, H. *A New Fluoroelastomer Derived From Tetrafluoroethylene and Propylene*. Rubber Chemistry and Technology, v. 50, 1977, p. 403.
2. FLORY, P. J. *Principles of Polymer Chemistry*. Cornell University Press, Ithaca, New York, 1953, p. 183.
3. STIVERS, D. A. *Fluoroelastomers* in the Vanderbilt Rubber Handbook, R. O. Babbitt, ed., R. T. Vanderbilt Co., Inc., Norwalk, CT, 1978, p. 245.

Table 1. FLUOROELASTOMERS

Polymer Commercial Name	United States Supplier	Weight Percent Fluorine	Chemical Structure
Aflas	Xenox/3M*	53.5	$ \begin{array}{cccc} \text{F} & \text{F} & \text{H} & \text{CH}_3 \\ & & & \\ -\text{C}- & \text{C}- & \text{C}- & \text{C}- \\ & & & \\ \text{F} & \text{F} & \text{H} & \text{H} \end{array} $
Fluorel	3M	71.0	$ \begin{array}{cccc} \text{H} & \text{F} & \text{F} & \text{F} \\ & & & \\ -\text{C}- & \text{C}- & \text{C}- & \text{C}- \\ & & & \\ \text{H} & \text{F} & \text{F} & \text{CF}_3 \end{array} $
Viton	Du Pont	71.0	$ \begin{array}{cccc} \text{H} & \text{F} & \text{F} & \text{F} \\ & & & \\ -\text{C}- & \text{C}- & \text{C}- & \text{C}- \\ & & & \\ \text{H} & \text{F} & \text{F} & \text{CF}_3 \end{array} $
Eypel F	Ethyl	54.0	$ \begin{array}{c} \text{O} \text{ CH}_2\text{CF}_3 \\ \\ -\text{P} = \text{N}- \\ \\ \text{O} \text{ CH}_2(\text{CF}_2\text{CF}_2)_{1-3}\text{H} \end{array} $
Silastic	Dow Corning	36.5	$ \begin{array}{c} \text{CH}_3 \\ \\ -\text{Si}-\text{O}- \\ \\ \text{CH}_2\text{CH}_2\text{CF}_3 \end{array} $

*3M Industrial Chemical Products Division, 3M Center, Bldg. 223-GS-04, St. Paul, MN 55144-1000. When this work began, Xenox, Inc. was the U.S. supplier. Currently, 3M performs that function.

Table 2. TYPES OF AFLAS
(An epoxy fluoropolymer has also been prepared; discussed in the section on compounding.)

Product Designation Xenox/3M	Comments
150 E / FA-150 E	Lowest Molecular Weight for Wire & Cable
150 P / FA-150 P	General Purpose
150 L / FA-150 L	Process Aid for Chemical Tank Linings
100 P*	Not Heat Treated (White Samples in This Report)
100 H / FA-100 H	Highest Molecular Weight, Replaced 100 P
100 S / FA-100 S	Improved Compression Set
200*	Resistant to Hydraulic Fluids Contains Double Bonds

*Not marketed by 3M, July 1987

Table 3. GENERAL PROPERTIES OF FLUOROELASTOMERS

Property	Viton	Fluorel	Aflas
Specific Gravity	1.80 - 1.86*	1.80 - 1.87*	1.5 - 1.6*
Hardness (Shore A)	50 - 95	50 - 95	60 - 95
Tensile Strength (psi)	2000	1300 - 2700	2000 - 3200
Elongation (%)	150 - 300	100 - 500	55 - 400
Compression Set (B) 392°F (200°C) ASTM D 395	50% (1000 hr) 10% - 70% (70 hr)	50% - 70% (1000 hr) 10% - 30% (70 hr)	30% (70 hr)
Brittle Point	-25°F - 75°F	-35°F	-55°F
Maximum Temperature Continuous Operation	450°F	400°F	400°F
Minimum Temperature Dynamic Applications	-23°C (-10°F)	-40°F (Seals)	†
Static Applications	To Brittle Point	†	†

*Gumstock

†No data

Table 4. THERMAL AND ELECTRICAL PROPERTIES OF FLUOROELASTOMERS

Property	Viton	Fluorel	Aflas
Specific Heat	0.395	*	*
Coefficient of Linear Expansion	$88 \times 10^{-6}/^{\circ}\text{F}$ $16 \times 10^{-5}/^{\circ}\text{C}$	*	*
DC Resistivity (ohm-cm)	2×10^{13}	2×10^{13}	*
Volume Resistivity (ohm-cm 500 Volts DC)	*	*	3.0×10^{16} , 70°F 1.7×10^{13} , 392°F
Dielectric Constant	10 100 Hz, 25°C	11.4 100 Hz, 25°C	2.6 60 Hz, 21°C
Dissipation Factor	0.05	0.0125	*
Dielectric Strength (Volts/mil)	500	630	580

*No data

Table 5. CHEMICAL RESISTANCE OF FLUOROELASTOMERS

	Viton	Fluorel	Aflas
Excellent	Oils Fuels Lubricants Most Mineral Acids	Automotive Fuels Hydrocarbons Aircraft Fuels and Oils Hydraulic Fluids Certain Chlorinated Hydrocarbons (Chloroform)	Acids Animal/Vegetable Oils Bases Brake Fluids Hydraulic Fluids (Phosphate Esters) Stream/Water/Brine
Good	Aliphatic and Aromatic Hydrocarbons	Aromatic Solvents Polar Solvents Aqueous Acids Water/Salt Solutions Dilute Alkali Oxidative Environments	Alcohol Amines Oils and Lubricants Oxidizing Agents
Poor	Ketones Esters Amines Strong Bases Ethers	Strong Caustic Ammonia Ketones Certain Polar Solvents	Benzene Fuels Ketones Chloroform

The properties in Table 3 appear to be quite comparable, except that the specific gravity is measured on compounded samples and, so, is quite variable. The specific gravity of Aflas materials is significantly lower than that of the other two elastomers, due to the lower fluorine content. This is often interpreted as resulting in a cost saving, since the polymers are purchased by weight. One of the shortcomings of fluorocarbon elastomers is their low temperature properties. A comparison of the use temperatures of elastomers is shown in Figure 1. Since fluorocarbon elastomers typically have high glass transition temperatures compared to other elastomers, their low temperature use is limited to static applications where chemical resistance is required. Where there is data to compare in Table 4, the major difference is in the dielectric constant. That of Aflas is considerably lower, which is not surprising considering the insulating character of propylene. The chemical resistance of the three elastomers is shown in Table 5. Viton and Fluorel seem to exhibit very similar chemical resistance, while Aflas differs. According to the manufacturer, Aflas shows excellent resistance to acids and bases, whereas the other polymers show poorer resistance to those substances. Another difference seems to be in resistance to halogenated solvents, Aflas showing poor resistance to chloroform, while Fluorel shows excellent resistance to chloroform.

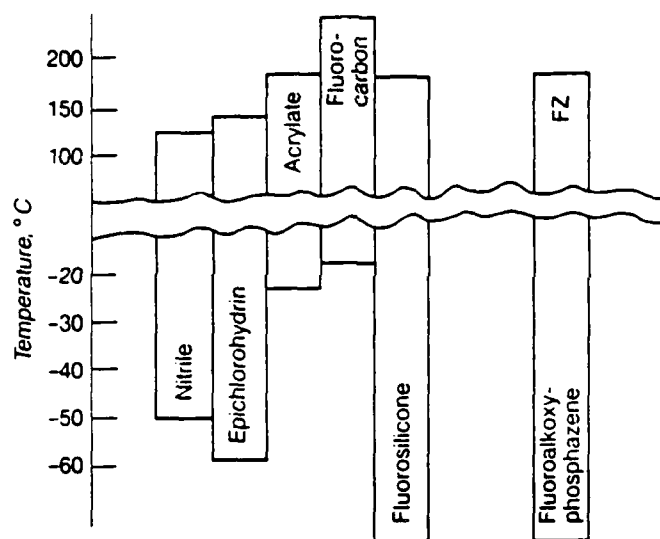


Figure 1. Service temperature range for oil/fluid resistant elastomers. [Reprinted from H. R. Penton, Kaut. & Gummi Kunst. (1986), with permission.]

MATERIALS

Samples of uncompounded Aflas were provided by Xenox, Inc. These include heat-treated 150 E, 150 P, 100 H, and 100 S, which are brown in color. A nonheat-treated sample, 100 P, was also included, which was white in color. Samples of poly(vinyl fluoride), poly(vinylidene fluoride), polytetrafluoroethylene, and propylene were obtained from Chem Services, Inc. An Aflas 150 P compound and a Fluorel compound were obtained from Smithers Scientific Services, Inc. The formulations are shown in Tables 6 and 7.

The remaining sections of this report will discuss the experimental technique used and the results of the experiments.

Table 6. RECIPE FOR FLUOREL FLUOROELASTOMER

Ingredient	PHR
Fluorel 2174*	80
Fluorel 2145	20
Carnauba Wax	3
N-990 Carbon Black	30
Maglite D (MgO) [†]	6
Calcium Hydroxide	2.5
	135.5

*Incorporated cure gum

[†]Supplied by C. P. Hall Co. or Calgon Corporation

Table 7. RECIPE FOR AFLAS FLUOROELASTOMER

Ingredient	PHR
Aflas 150 P	100
N-990 Carbon Black	50
Carnauba Wax	2
Diak #7*	7
Luperco 101XL [†]	9
	169

*Supplied by Du Pont Co.

[†]Supplied by Harwick Chemical Corp. or Pennwalt Corp.

SOLUBILITY

For several types of analysis, solutions of the uncompounded elastomer are required. Mixtures of Freon TF and ethyl acetate were recommended by the supplier, but the best solvent for Aflas seems to be tetrahydrofuran. Very dilute solutions of the 150 and 100 series samples were prepared, so that they might be analyzed by size exclusion chromatography (SEC), also called gel permeation chromatography. This would provide an estimate of the molecular weights relative to the molecular weights of polystyrene standards. Before SEC, samples are filtered, usually through a 0.45 μ or a 1.0 μ filter. These solutions were not filterable, indicating that some polymer gel remained even after stirring and gentle heating for several days. No reliable estimate of molecular weight could be obtained using SEC for such samples. Solutions in deuterated tetrahydrofuran were used for nuclear magnetic resonance experiments (NMR).

NUCLEAR MAGNETIC RESONANCE

Proton and carbon NMR spectra were run on a Varian XL-200 instrument. The ¹H spectrum of sample 100 H is shown in Figure 2. The peaks at 3.55 ppm and 1.7 ppm are due to the THF solvent. The peak at 1.26 ppm is due to the methyl group of propylene, and multiplets at 2.03, 2.50, and 2.60 are due to the methine and methylene protons, respectively. The spectrum is consistent with the alternating copolymer structure stated by the manufacturer. The peaks are broadened, but are consistent with the theoretical spectrum from isolated -CH₂CH(CH₃)- repeat units, a doublet for the methylene group, a multiplet for the

methine, and a doublet for the methyl group. The integration of the spectrum does not fit the structure and no explanation is offered for this behavior. A research paper on the fluorine NMR spectra of the polymers concludes that the polymers have an alternating structure.¹ The proton decoupled ^{13}C NMR spectrum for the same polymer is shown in Figure 3. The multiplets at 25 ppm and 67.4 ppm are due to the THF. The singlet at 14.6 ppm is due to the methyl carbon. At 31.1 ppm, are overlapping resonances due to the carbon of the methylene and methine groups. Between 110 ppm and 130 ppm are two overlapping triplets of triplets, due to coupling of fluorine with the carbon atoms of the tetrafluoroethyl repeat unit. This spectrum is also consistent with the alternating structure suggested by the manufacturer, suggesting isolated tetrafluoroethylene and propylene units.

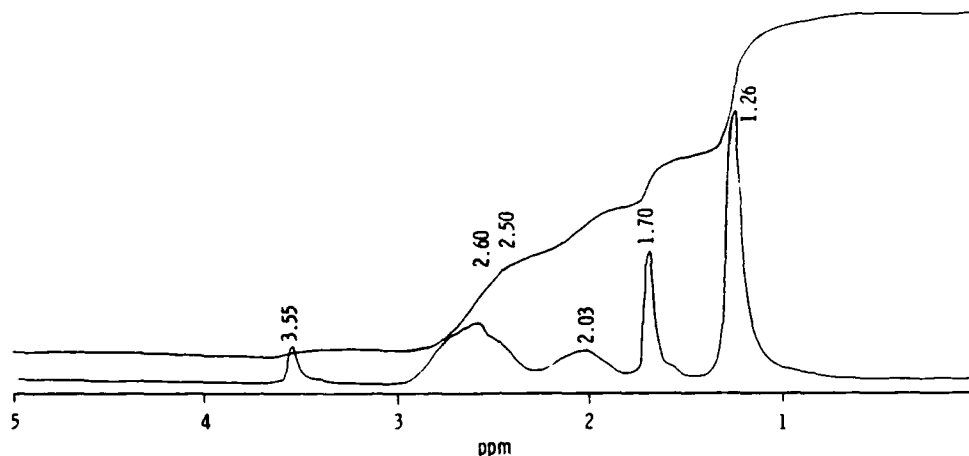


Figure 2. Proton NMR spectrum of Atlas 100 H.

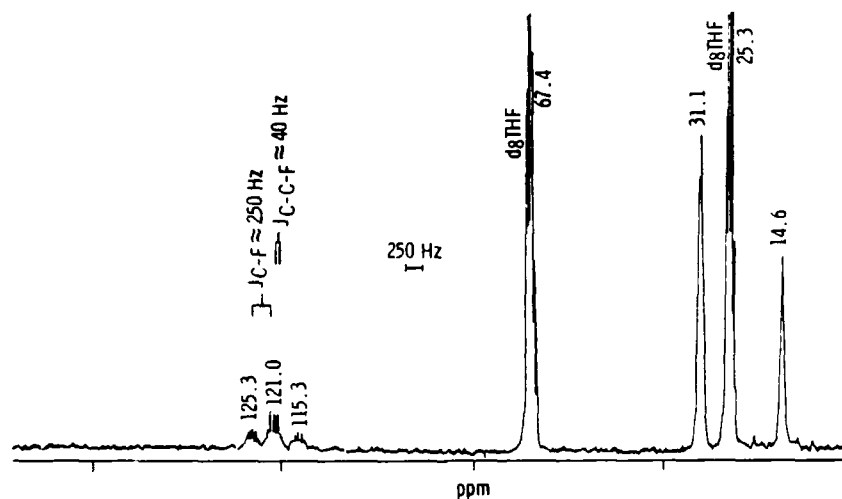


Figure 3. Proton decoupled ^{13}C NMR spectrum of Atlas 100 H.

INFRARED SPECTROSCOPY

Infrared spectra were run on a Perkin-Elmer 1500 spectrophotometer equipped with a Wilks Scientific model 37 heating cell. The sample compartment was kept in flowing nitrogen. Aflas 100 P was dissolved in tetrahydrofuran and cast as a thin film on a KBr salt plate. The spectrum is shown in Figure 4. The spectrum contains peaks in the region of 2840 to 3000 cm^{-1} due to methylene and methyl group stretching, weak doublets in the region 1350 to 1475 cm^{-1} due to other motions of the methylene, and methyl groups and several strong peaks in the region 1000 to 1300 cm^{-1} due to C-F stretching in the CF_2 and CF_3 groups.⁴ The sample was also heated to 350°F for thirty minutes in order to observe double bond formation, if any. This Aflas sample was white and not heat treated, whereas the other samples were heat treated and dark brown in color. Presumably, the heat treatment was used with the hope of introducing double bonds into the polymer. The double bonds could be used in a vulcanization process which would be much more efficient than the peroxide vulcanization available for use in polymers which contain no double bonds. Double bond formation was undetected in the heated sample. This explains why Aflas 200 has been developed which contains a small amount of an unsaturated third monomer for crosslinking.⁵⁻⁷ (See Table 2.) This material is apparently not available from 3M.

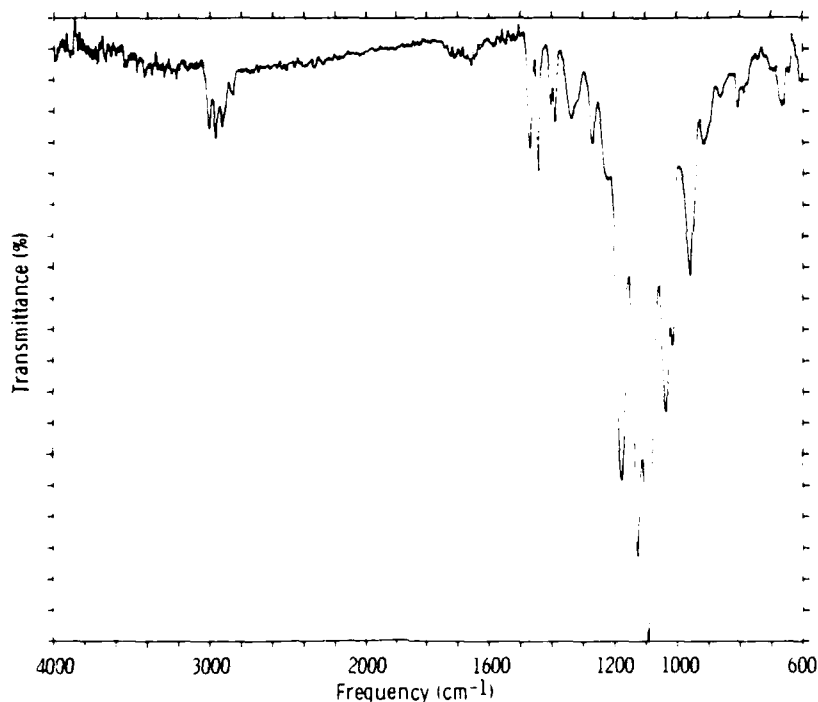


Figure 4. Infrared spectrum of Aflas 100 P. Sample cast from tetrahydrofuran solution onto a KBr salt plate and dried.

4. DYER, J. R. *Applications of Absorption Spectroscopy of Organic Compounds*. Prentice-Hall, Englewood Cliffs, NJ, 1965, p. 30.
5. KODAMA, S., WACHI, H., ITO, Y., MOROZUMI, M., and KOJIMA, G. *Unique Fluoroelastomers for Diverse Industrial Uses with Harsh Environments*. International Rubber Conference 1985 Program, Kyoto, October 15-18, p. 820.
6. KOJIMA, G., and WACHI, H. *A New Tetrafluoroethylene-Propylene Based Fluoroelastomer with Improved Low Temperature Properties*. International Rubber Conference 1985 Program, Kyoto, October 15-18, p. 242.
7. HULL, D. E., KOJIMA, G., and WACHI, H. *New Types of Fluoroelastomers Provide Improved Resistance to Some Automotive Media*. 133rd American Chemical Society Rubber Division Meeting, Dallas, April 1988, paper no. 17.

NEUTRON ACTIVATION ANALYSIS

The percent fluorine in the Aflas polymers was measured using neutron activation analysis. A 14.7 MeV neutron generator from Kaman Sciences was used, together with an ORTEC ADCAM multichannel analyzer and an intrinsic germanium detector. Polytetrafluoroethylene pellets were used as a standard. The results are shown in Table 8. For a strictly alternating copolymer with a repeat unit of $-\text{CF}_2\text{CF}_2\text{CH}(\text{CH}_3)\text{CH}_2-$ the weight percent fluorine is 53.5%. All of the samples contain more fluorine than the theoretical, indicating that more tetrafluoroethylene monomer is incorporated. Evidence for this has also been obtained from fluorine NMR.¹ The lower molecular weight samples 150 E and 150 P incorporate more fluorine, indicating less alternating structure for those materials. These were the earliest materials marketed. This data suggests that the manufacturer was able to improve the polymerization process, perhaps by choice of a catalyst which would increase the alternation of the monomers in the polymer chain. Also note that if adjacent propylene moieties occurred rather than adjacent tetrafluoroethylene units, much poorer properties would result.

Table 8. NEUTRON ACTIVATION ANALYSIS RESULTS

Aflas Sample	% Fluorine
150 E	62.7
150 P	64.7
100 H	57.0
100 P	61.3
100 S	57.3
Theoretical Value	53.5

THERMOGRAVIMETRIC ANALYSIS

Thermogravimetric analyses (TGA) were performed with a Perkin-Elmer TGS-2 Thermogravimetric System. Dynamic scans were conducted at a rate of 10°C/min. Isothermal analyses were conducted by heating at 40°C/min to 350°C and holding for the balance of the stated time. The atmosphere was flowing dry air at a rate of 50 ml/min. Data was acquired using a Bascom-Turner M/8110 Intelligent Recorder, stored on disk and then plotted.

Figure 5 shows dynamic TGA curves for the tetrafluoroethylene-propylene copolymer and polytetrafluoroethylene. Significant weight loss begins at approximately 425°C for the copolymer and at 525°C for the homopolymer. The results show one reason why Aflas can be used in continuous service at 400°C. The weight loss is one measure of polymer degradation. Isothermal aging results are shown in Figure 6. The copolymer is compared to similar polymers containing more hydrocarbon character. It is interesting that the Aflas is more stable than the other materials, even though it can form a stable tertiary free radical during degradation. This is not possible for the other fluoropolymers, poly(vinylidene fluoride) and poly(vinyl fluoride), but is possible for the comparatively unstable polypropylene. Note that Aflas does lose a small amount of weight, which will be discussed in the next section.

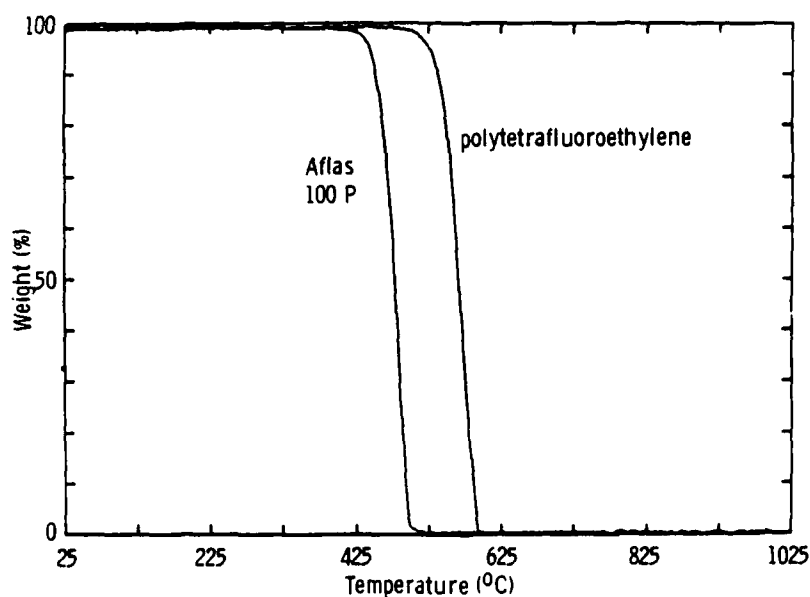


Figure 5. Dynamic thermogravimetric analysis curves in air.
Scan rate is $10^{\circ}/\text{min}$.

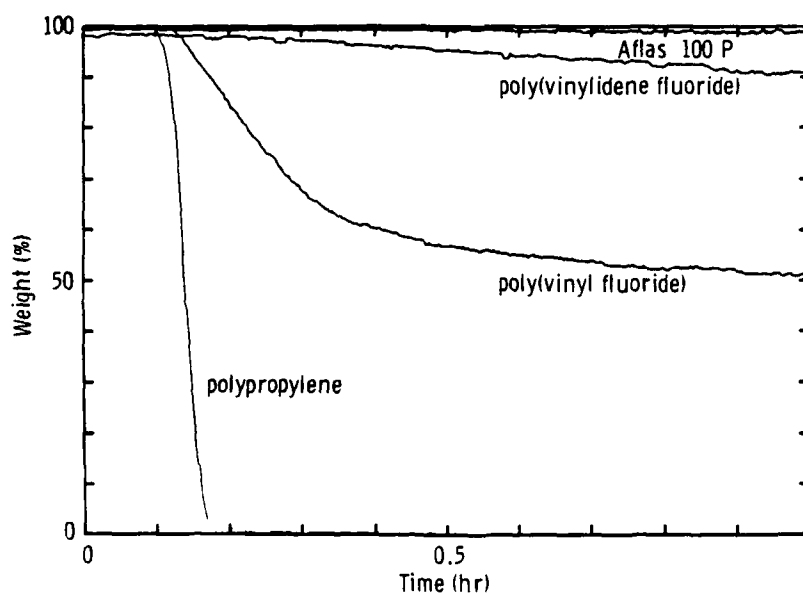


Figure 6. Thermogravimetric analysis isothermal aging
curves at 350°C in air.

PYROLYSIS-GAS CHROMATOGRAPHY-MASS SPECTROSCOPY

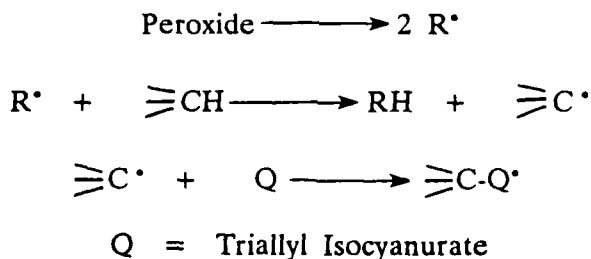
Pyrolysis-gas chromatography-mass spectroscopy was also used to study the thermal treatment of the 100 P sample. The sample was analyzed using a Hewlett-Packard 5996 GC/MS. A 5.0 mg sample was placed in a quartz tube holder and inserted into a platinum coin desorption probe. The probe was inserted into a pyrolysis-GC interface maintained at 260°C . The controller was manufactured by Chemical Data Systems, model number 122. The injection port of the GC was set at 280°C and the GC oven was set at -20°C . A 12 m, fused silica

capillary column of crosslinked dimethylsilicone stationary phase was used. Helium was used as the GC mobile phase. The desorption probe was ramped at 20°C/msec to 350°C and held at that temperature for 20 sec. The GC oven was ramped at 10°C/min to 300°C and held for 10 min. The quadropole mass spectrometer, operating in the electron impact mode at 70 eV, was scanned from 10 to 800 amu at a rate of 1 sec/scan. Data acquisition, storage, and reduction was performed using a Hewlett-Packard 1000 E-series computer, running RTE-6/VM software.

Only hydrocarbons were observed in the mass spectrometer, mostly fragments of twelve and fourteen carbons. This suggests that the propylene oligomerizes under the polymerization conditions, and that the heat treatment is, in fact, useful in removing the low molecular weight materials. They would have a negative effect on the final properties of the compounded elastomer.

COMPOUNDING

It should be noted that Aflas, Viton, and Fluorel are actually gummy polymers in the pure state, and that they become elastomeric only on compounding, which is a crosslinking process. Most forms of Aflas contain no double bonds and can, therefore, only be cured by electron beams or by peroxides.⁸ In the cure recipe shown in Table 7, Luperco 101XL is a peroxide curing agent and Diak #7 is triallyl isocyanurate (TAIC), a coagent. The Diak #7 contains three double bonds per molecule and so promotes crosslinking once free radicals are formed.⁹



A high vinyl 1,2-polybutadiene like Ricon 153 (Colorado Chemical Specialties) can be used in addition to TAIC.¹⁰ It accelerates the cure and improves the extrusion resistance of molded parts.

In order to solve the problems created by the lack of double bonds or other sufficiently reactive sites in Aflas, the manufacturer has investigated the possibility of incorporating epoxy groups as cure sites in Aflas-like materials. The results have been published. This polymer could be cured with blocked hexamethylene diamine using acid salts of the diamine, and showed mechanical properties and chemical resistance similar to the TFE/P polymer cured by peroxides.¹¹

The remainder of this report will discuss results obtained using the compounded elastomer.

8. KOJIMA, G., and WACHI, H. *Vulcanization of a Fluoroelastomer Derived from Tetrafluoroethylene and Propylene*. Rubber Chemistry and Technology, v. 51, 1978, p. 940.
9. CORAN, A. Y. *Vulcanization*. Ch. 7 in Science and Technology of Rubber, F. R. Eirich, ed., Academic Press, NY, 1978, p. 327-328.
10. 3M Industrial Chemical Products Division, Product Literature on Aflas, 1987.
11. KOJIMA, G., KOJIMA, H., MOROZUMI, M., WACHI, H., and HISASUE, M. *Vulcanization and Vulcanizate Properties of a Fluoroelastomer Containing Epoxy Groups as Cure Sites*. Rubber Chemistry and Technology, v. 54, 1981, p. 779.

DIFFERENTIAL SCANNING CALORIMETRY

Dynamic differential scanning calorimetry (DSC) was performed on a Perkin-Elmer DSC-2 instrument in the range of 150°K to 300°K. The base temperature was maintained with liquid nitrogen, and the instrument was purged with helium. Samples weighing 10 mg to 15 mg were encapsulated in standard aluminum pans and heated at a rate of 20°/min. The resulting curves were analyzed using a Perkin-Elmer Thermal Analysis Data Station. The results are shown in Table 9.

The Aflas copolymers exhibit T_g s near 0°C, which is improved by compounding to -7°C, whereas the Fluorel elastomers have T_g s near -25°C. The low temperature use is perhaps extended 20°C.

Table 9. THERMAL PROPERTIES AND HARDNESS OF AFLAS

Sample	DSC T_g (°C)	TMA Transitions (°C)	Hardness (Shore A)
Aflas			
150 E	0	-	-
150 P	1	4, 48	-
100 H	2	-	-
100 P	2	-	-
Compounded*	-7	4	83
Fluorel			
2145	-24	-11, 31	-
2174	-24	-20, 37	-
Compounded*	-21	-14	78

*Compounded samples are 0.040" thick

HARDNESS

The Shore A hardness determined using ASTM D 2240-75 (shown in Table 9) is representative, but can vary considerably depending on compounding ingredients.

THERMOMECHANICAL ANALYSIS

Thermomechanical analysis (TMA) was performed on a Perkin-Elmer thermomechanical analyzer, TMS-1 with a Perkin-Elmer model UU-1 temperature program controller. Results were recorded on a Soltec chart recorder, model 1242. The samples were 1.0-mm thick. The weight used on the TMA probe was 20 g. The heating rate was 20°C/min and the chart speed was 20 mm/min. Samples were heated in a helium atmosphere from -100°C to the softening point of the samples. The results are shown in Table 9.

The values of the TMA transitions are consistent with the results of DSC, and include final softening temperatures for some of the uncompounded polymers 10° to 30° above room temperature. The compounded elastomers did not show final softening in the region scanned up to about 270°C.

TENSILE PROPERTIES

Standard samples were prepared and tested using ASTM methods for tensile properties D1708-66 using a microdie. Stress-strain curves for the compounded elastomers are shown in Figures 7, 8, and 9, each one for two samples of the same thickness. The stress-strain properties depend, to some extent, upon the compounding. For the 10 and 20 mil samples, the Aflas compound is the stiffer, stronger material, but the 40 mil samples exhibit the opposite. The properties of the Aflas are so poor, that a mishap during the compounding operation is suspected.

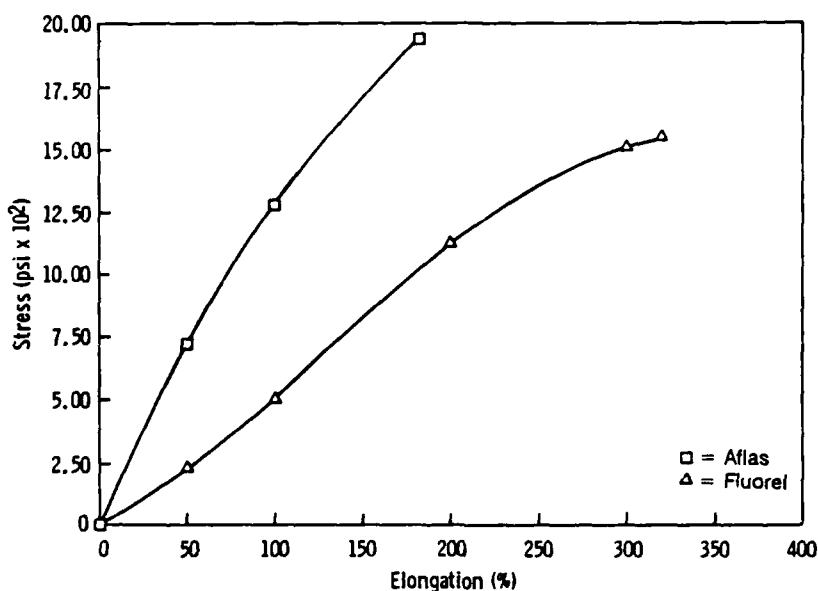


Figure 7. Stress-strain curves for 0.010" samples.

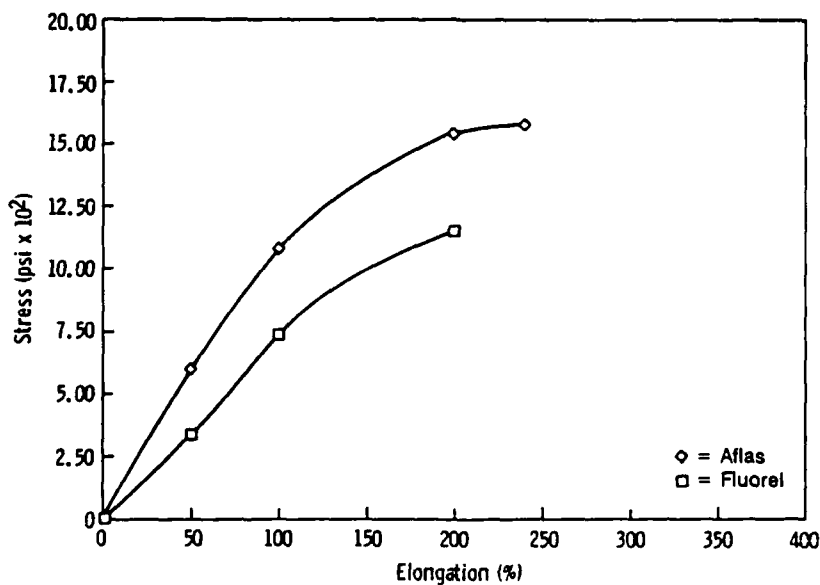


Figure 8. Stress-strain curves for 0.020" samples.

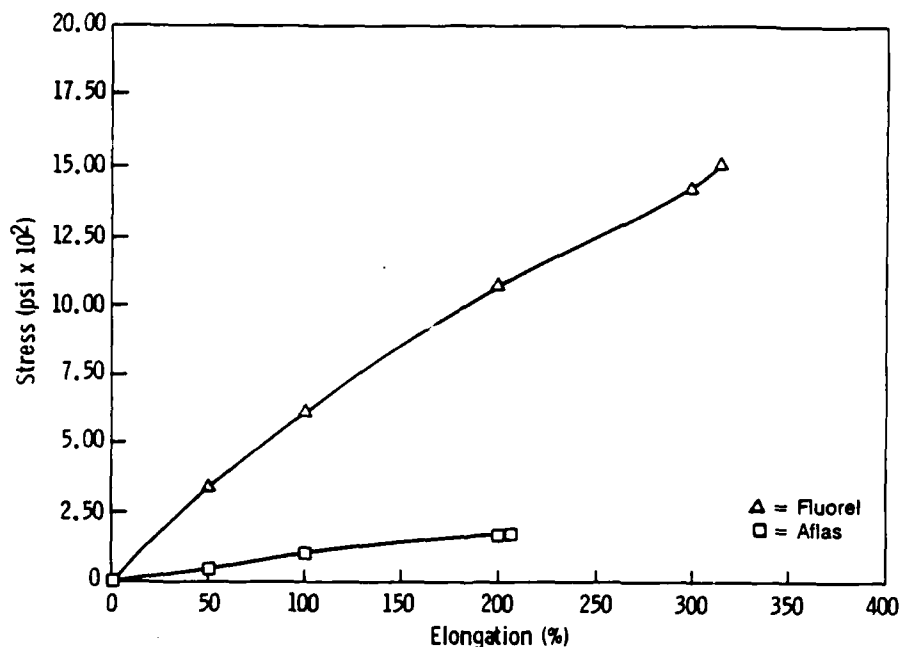


Figure 9. Stress-strain curves for 0.040" samples.

DYNAMIC MECHANICAL ANALYSIS

Dynamic mechanical analysis was performed on a Rheometrics dynamic spectrometer, RDS-7700. The samples were cut to 13 mm by 64 mm and were 1.0-mm thick. Measurements were taken at 10°C increments, with an equilibration time of 2 min at each temperature. The temperature range studied was from -50°C to 200°C with a strain setting of 1.0 for Aflas, 1.2 percent for Fluorel, and a rate of 6.28 radians per second.

The dynamic mechanical spectra are shown in Figures 10 and 11. The polymers exhibit remarkably similar behavior in the region where they can be compared. The peaks in the tan delta curves occur at different temperatures, but are similar in magnitude. Comparing the stiffness at -50°C, the storage moduli, G' , are 1.6×10^{10} and 1.7×10^{10} dynes/cm², respectively, for Aflas and Fluorel compounds, respectively. At 100°C, values of 7.6×10^7 and 6.4×10^7 were obtained. This suggests that the materials would behave similarly at the temperatures mentioned.

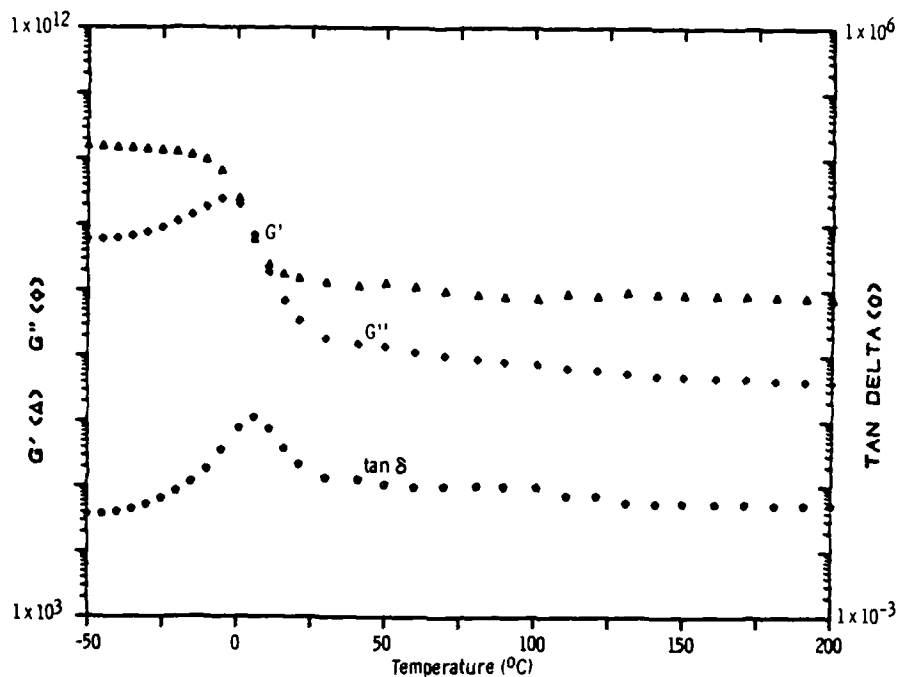


Figure 10. Dynamic mechanical spectrum for Atlas.
Sample thickness = 1.0 mm (0.040").

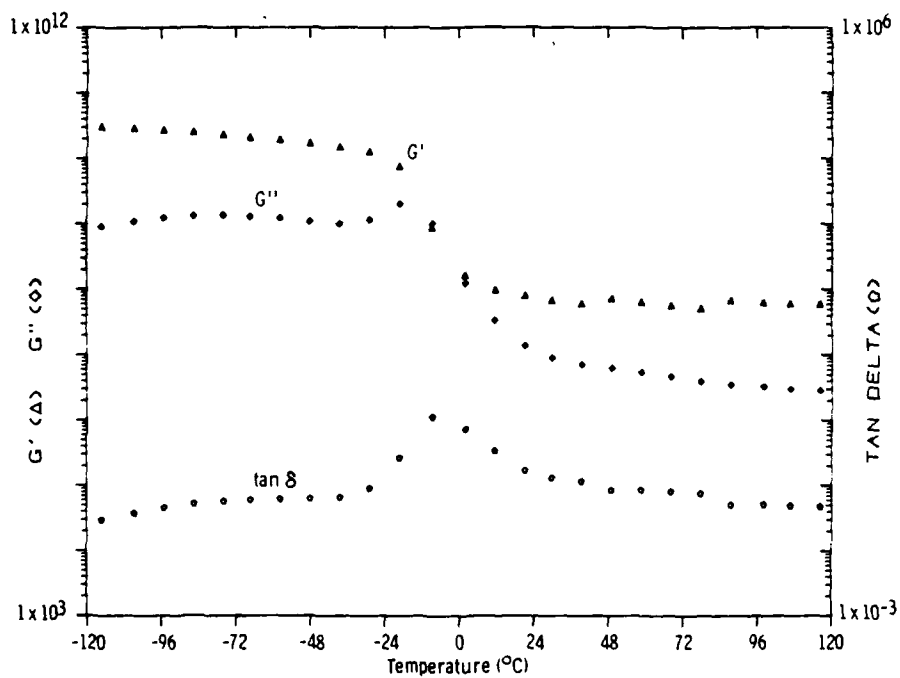


Figure 11. Dynamic mechanical spectrum for Fluorel.
Sample thickness = 1.0 mm (0.040").

IMMERSION TESTING

Immersion testing is the method used to screen many types of polymers for chemical resistance. A 1-inch disk, in the present case, 40-mils thick, of the compounded elastomers are immersed in a liquid of interest at approximately 22°C. The samples are removed from the jar, blotted dry, and weighed at regular intervals, more often at the beginning of the experiment and less often as the experiment progresses. When the weight of the sample plus sorbed liquid reaches a constant value, the experiment is ended.

The results of this experiment are shown in Table 10. The Aflas performed better in diisopropyl methyl phosphate (DIMP), absorbing much less in a longer period of time. If one considers total weight absorbed, it also performs better in dichloropentane (DCP), but only slightly. Weight gains of 6 to 14 percent are quite small in tests of this kind on elastomers. The results of this test are a little surprising when one views the data in Table 5. Aflas performs poorly in chloroform, but Fluorel has excellent resistance to chloroform. Not included in Table 5 are the chemical resistance data for other chlorinated hydrocarbons, which show mixed results according to the suppliers. Aflas has poor chemical resistance to carbon tetrachloride and trichloroethylene, good resistance to methylene chloride, and excellent resistance to dichlorobutane, the most similar compound tested to DCP. Fluorel has poor resistance to methylene chloride, excellent resistance to carbon tetrachloride and trichloroethylene, however, dichlorobutane was not tested. Ethylene dichloride is the most like DCP of the halogenated solvents tested and shows excellent resistance. The interactions of the chlorinated solvents and the fluorinated polymers are complex and difficult to predict.

Table 10. IMMERSION RESULTS ON COMPOUNDED ELASTOMERS

Smithers Rubber Compound	Maximum Weight Gain (%) When Immersed in	
	DCP*	DIMP†
Aflas	6.3 (145 hr)	23 (78 hr)
Fluorel	14 (3200 hr)	199 (48 hr)

*DCP is 1,5-dichloropentane

†DIMP is diisopropyl methyl phosphonate

From the results discussed in the preceding paragraph, one would choose Aflas for use in an environment where chemical agents might be present, since the resistance of Aflas to both simulants was very good. Caution is always advised in using results of tests with simulants, because the only way to guarantee resistance to live agents is to test the polymeric materials with the live agents.

The NBC Materials Handbook¹² summarizes testing of polymeric materials with chemical agent decontaminants. Both Aflas and Viton compounds were tested and showed excellent resistance to decontaminating solutions. Very small volume changes occurred on immersion for 24 hours, and both elastomers retained their strength.

12. NBC Materials Handbook. Hughes Aircraft Co., Missile Systems Group, Canoga Park, CA, 1982, p. 3-16, 3-25.

APPLICATIONS

The earliest uses for fluoroelastomers was in O-rings. Solid and cellular products can be made by molding, extruding, or calendaring. Solutions can be used and coated fabrics can be prepared. There are automotive applications in hydraulic seals, automatic transmission seals, and carburetor needle valve tips. In aircraft, fluoroelastomers are used in seals where thermal cycling is required, and for exceptional lubricant and fuel resistance. Other uses include wire and cable coatings and in chemical process equipment for seals, gaskets, and transfer hoses.

CONCLUSIONS

Fluorocarbon elastomers are used in many applications where their chemical resistance at elevated temperatures is important. It is clear from the data presented here that in many ways (e.g., mechanical properties), Aflas is comparable to the older Viton and Fluorel products. There are also applications where the unique properties of Aflas make it the polymeric material of choice for uses where its chemical resistance behavior is better than that of the more traditional materials. Need for a material with a low dielectric constant might lead to a choice of Aflas materials over the Viton or Fluorel materials.

ACKNOWLEDGMENTS

The author would like to thank David Bulpett for the mass spectroscopy, Michaeline Chen for the neutron activation analysis, Emily McHugh for thermal analysis and dynamic mechanical analysis, Gretchen Richard for the nuclear magnetic resonance spectroscopy, James Sloan for the infrared spectroscopy, Stanley Wentworth for the Thermogravimetric Analysis, and Anthony Wilde for providing the compounded samples and for the immersion test results.

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STUDIES OF AFLAS FLUOROELASTOMERS -
Catherine A. Byrne

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Technical Report MTL TR 89-69, July 1989,
19 pp-illus-tables.

This report includes studies of Aflas, a family of Japanese fluoroelastomers, and comparison of this material with two important fluoroelastomers manufactured in the United States. While Fluorel and Viton are prepared from only fluorinated monomers, Aflas is an alternating copolymer of tetrafluoroethylene and propylene. Because of the alternating character of the polymer, Aflas possesses many of the desirable properties of Fluorel and Viton and also has some unique properties which might make it useful in applications where the other two are inferior. Differences noted in this work include dielectric constants in the range of 10 to 11 for Fluorel and Viton and 2.6 for Aflas. The glass transition temperature of a Fluorel compound was -21°C compared to that for an Aflas compound of -7°C. This means that Fluorel could be used in dynamic applications for about 10°C lower than Aflas. This could be significant because fluoroelastomers are known to have limited applications at low temperatures anyway. Chemical resistance of the elastomers is different for certain classes of chemicals. Aflas is reported to have better resistance to aqueous bases than that of the other two. Immersion testing in two chemical agent simulants was performed on both Fluorel and Aflas compounds. Aflas performed much better in diisopropyl methyl phosphonate, but the two elastomers both performed well in 1,5-dichloropentane. The manufacturers report varied resistance to chlorinated solvents. Aflas has poor resistance to carbon tetrachloride and trichloroethylene and excellent resistance to dichlorobutane, the closest to dichloropentane tested. Fluorel is reported to have excellent resistance to the first two and dichloroethylene, however the dichlorobutane was not tested.

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Technical Report MTL TR 89-69, July 1989,
19 pp-illus-tables.

This report includes studies of Aflas, a family of Japanese fluoroelastomers, and comparison of this material with two important fluoroelastomers manufactured in the United States. While Fluorel and Viton are prepared from only fluorinated monomers, Aflas is an alternating copolymer of tetrafluoroethylene and propylene. Because of the alternating character of the polymer, Aflas possesses many of the desirable properties of Fluorel and Viton and also has some unique properties which might make it useful in applications where the other two are inferior. Differences noted in this work include dielectric constants in the range of 10 to 11 for Fluorel and Viton and 2.6 for Aflas. The glass transition temperature of a Fluorel compound was -21°C compared to that for an Aflas compound of -7°C. This means that Fluorel could be used in dynamic applications for about 10°C lower than Aflas. This could be significant because fluoroelastomers are known to have limited applications at low temperatures anyway. Chemical resistance of the elastomers is different for certain classes of chemicals. Aflas is reported to have better resistance to aqueous bases than that of the other two. Immersion testing in two chemical agent simulants was performed on both Fluorel and Aflas compounds. Aflas performed much better in diisopropyl methyl phosphonate, but the two elastomers both performed well in 1,5-dichloropentane. The manufacturers report varied resistance to chlorinated solvents. Aflas has poor resistance to carbon tetrachloride and trichloroethylene and excellent resistance to dichlorobutane, the closest to dichloropentane tested. Fluorel is reported to have excellent resistance to the first two and dichloroethylene, however the dichlorobutane was not tested.